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Results of an International Round-Robin for Tensile Creep Rupture of Silicon Nitride

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Fourteen laboratories participated in an interlaboratory study to establish the within- and between-laboratory repeatability of tensile creep rupture of silicon nitride. In air at 1375°C at 200 MPa, the times to failure ranged over a factor of 50, and the minimum creep rates ranged over a factor of 20. Despite these large ranges, taken individually, no one laboratory stands out from any other; all produced equally acceptable data. Consumers of silicon nitride tensile creep data must accept this magnitude of variability in reported creep data. The wide variety of specimen shapes and sizes, gripping systems, extensometry techniques, and temperature measurement strategies makes it impossible to assign definitively the root cause of the variability. However, there was a significant specimen size effect. As a group, the small-diameter specimens lasted roughly five times longer and crept three times more slowly than the large-diameter buttonhead specimens. A possible interpretation of the origin of this difference is that the oxidizing conditions affected more of the volume of the small specimens during the test.

I. Introduction

During the past 10 years, silicon nitride has achieved the status of an engineering material for gas turbines[1,2] as well as for other high-temperature applications, such as automotive valves.[3] It has done so by virtue of its excellent strength, toughness, and high-temperature creep resistance. To use laboratory-generated creep data for engineering design, as opposed to simple scientific comparison, the user must consider several questions. What is maximum deviation of these data from the true value? Does the variability in reported values indicate intrinsic variability in the material? Does the variation result from the test technique of the laboratory? Interlaboratory comparisons, or round-robins, can help answer these questions. During the 38 years since the first tensile creep test of silicon nitride,[4] there has been only one, very small interlaboratory study.[5] This article reports the results of the first large-scale, round-robin test for tensile creep rupture of silicon nitride, and it provides a first attempt to answer these questions for silicon nitride.

II. Testing Condition Background

Fourteen laboratories, listed in Table I, participated in the study. Figure 1 shows schematics of their specimens. Five laboratories tested large, buttonhead specimens, which were gripped outside the hot-zone of the furnace. Generally, these laboratories measured strain by capacitive, contact extensometry, such as Liu and Ding[6] have described (denoted "contact" in Table I). Nine laboratories tested much smaller pin- or edge-loaded specimens. All these specimens were gripped inside the hot-zone of the furnace. Most of these laboratories used some variant of a laser extensometry technique[7,8] to measure strain (denoted "laser/flags" in Table I). In laser[8] or electrooptical extensometry, flags attached to the specimen gauge length interrupt a light source. The shadow of the flags falls on a photograph–detector, and the change in position of the shadows is interpreted as the change in gauge length due to creep. The University of Dayton Research Institute uses a novel laser diffraction technique[9] that shares many advantages and limitations with the laser extensometry method. Many of the laboratories that tested the small specimens do not normally use the SR76 specimen, shown in Fig. 1, but found that it fitted in their load trains without modification. One exception was the Technical University of Hamburg–Harburg, which fabricated a shoulder-loaded specimen for their load train from existing SR76 pin-loaded specimens. Space does not permit discussion of the details of the extensometry and gripping systems of each laboratory: interested readers should consult the references listed in Table I.

This study used a later vintage of the same grade of gas-pressure-sintered silicon nitride† that the National Institute of Standards and Technology (NIST) had used in a preliminary, five-laboratory comparison in 1994,[5] as well as in a comparison of different specimen geometries,[14] and a large study of tensile creep rupture.[15] The gas-pressure-sintered silicon nitride came in the form of five billets, 19 mm thick and nominally 100 mm × 180 mm. As much as possible, each laboratory received specimens from each billet, but because each laboratory tested only three specimens, not every laboratory tested specimens from every billet. A ceramic machine shop in the United States fabricated all the specimens for European and U.S. participants. Japanese participants fabricated their own specimens from blanks machined from the original five billets.

The instructions to the participants were simple: test three specimens to failure in air at 1375°C under a 200 MPa load. A preliminary study of five specimens at NIST, conducted after the fabrication of the participants' specimens, established these conditions. They are a compromise between technologically interesting conditions, which favor low temperatures and high stresses, and those that produce reasonably short failure times at lower stresses, which enable more laboratories to participate. Before loading, each laboratory held the specimen under a 10 MPa load for 24 h, which is the usual NIST procedure. At the end of the testing campaign, the participants returned the broken specimens and the creep curves to NIST for further analysis. The round-robin followed ASTM Standard Practice E 691,[16] "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method."

III. Results

Figure 2 shows the individual creep curves by laboratory, plotted on identical axes. Laboratory 26 appears twice, because it

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†SN-88, NGK Insulators, Nagoya Japan. Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

Table I. Participants, in no Particular Order, Who Were Able to Complete at Least One Test

Participant	Laboratory	Extensometry	Specimen geometry	Reference†
Mitsuru Hattori	NGK Insulators, Ltd.	Crosshead displacement	NGK buttonhead	10
A. Wereszczak	Oak Ridge National Laboratory High Temperature Materials Laboratory	Contact	Buttonhead	11
Ken Liu	Oak Ridge National Laboratory Metals and Ceramics Division	Contact	Buttonhead	6
Mineo Mizuno	Japan Fine Ceramics Center	Contact	Buttonhead	
George Graves	University of Dayton Research Institute	Laser diffraction	Buttonhead	9
H. T. Lin	Oak Ridge National Laboratory Metals and Ceramics Division	Laser/flags	Pin-loaded SR76	12
William Luecke	National Institute of Standards and Technology Ceramics Division	Laser/flags	Pin-loaded SR76, SR51	7, 8
Junghyun Cho	Lehigh University Department of Materials Science	Laser/flags	Pin-loaded SR76	
Roger Cannon	Rutgers University Department of Ceramics	Laser/flags	Pin-loaded SR76	
Tatsuki Ohji	National Industrial Research Institute of Nagoya	Electro-optical/flags	Edge loaded	13
Jakob Kübler	Swiss Federal Laboratories for Materials Testing and Research (EMPA)	Contact	Pin loaded	
Jonathan Salem	NASA Glenn Research Center	Laser/flags	Pin loaded	
Dietmar Koch	University of Bremen Institute for Ceramic Materials and Components	Contact	Wedge grip	
Andreas Rendtel	Technical University of Hamburg–Harburg	Laser/flags	Edge-loaded modified SR76	70

†Published sources of more information on each technique.

tested two different specimen geometries—the 76 mm long SR76 and the 51 mm long SR51. The creep curves themselves exhibit the full range of creep behavior, from all primary to significant tertiary, sometimes with each behavior resulting in a single laboratory. Two-thirds (15/24) of the small specimens (those with cross sections $<1.2 \times 10^{-5}$ m^2) showed tertiary creep, but fewer than one-fourth (4/15) of the large specimens showed tertiary creep. As a group, the small specimens crept to ~1.5 times the strain of the large specimens.

Figure 3 summarizes the logarithms of the times to failure and minimum creep rates by laboratory. The times to failure vary by a factor of almost 50, and the minimum creep rates by 20. The

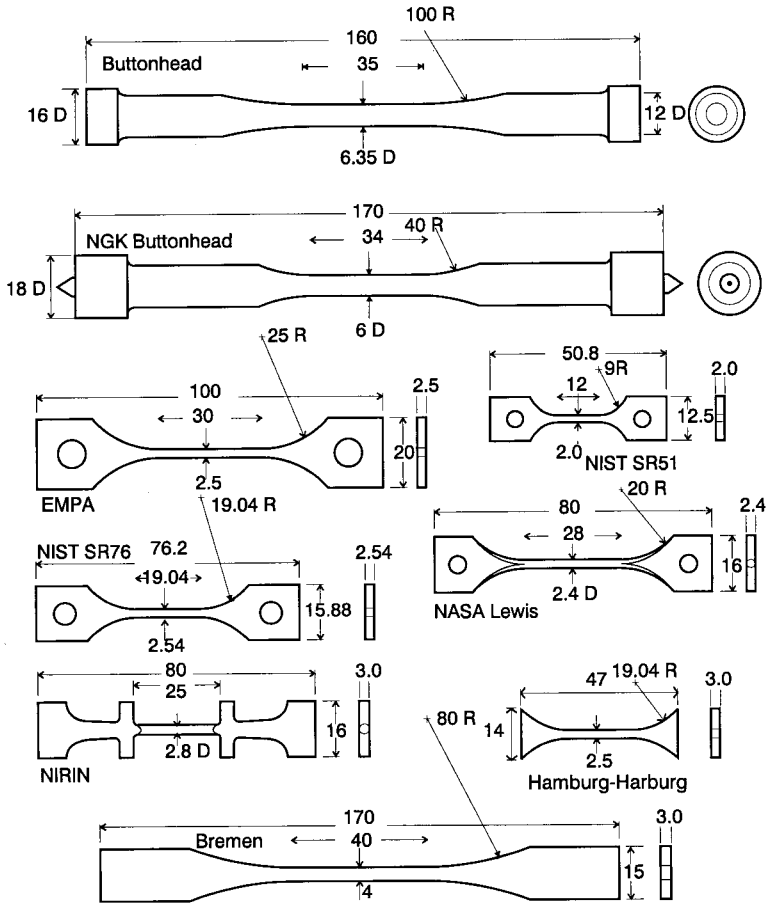

Fig. 1. Nine different specimens (drawn to scale) of this study. All dimensions are in millimeters.

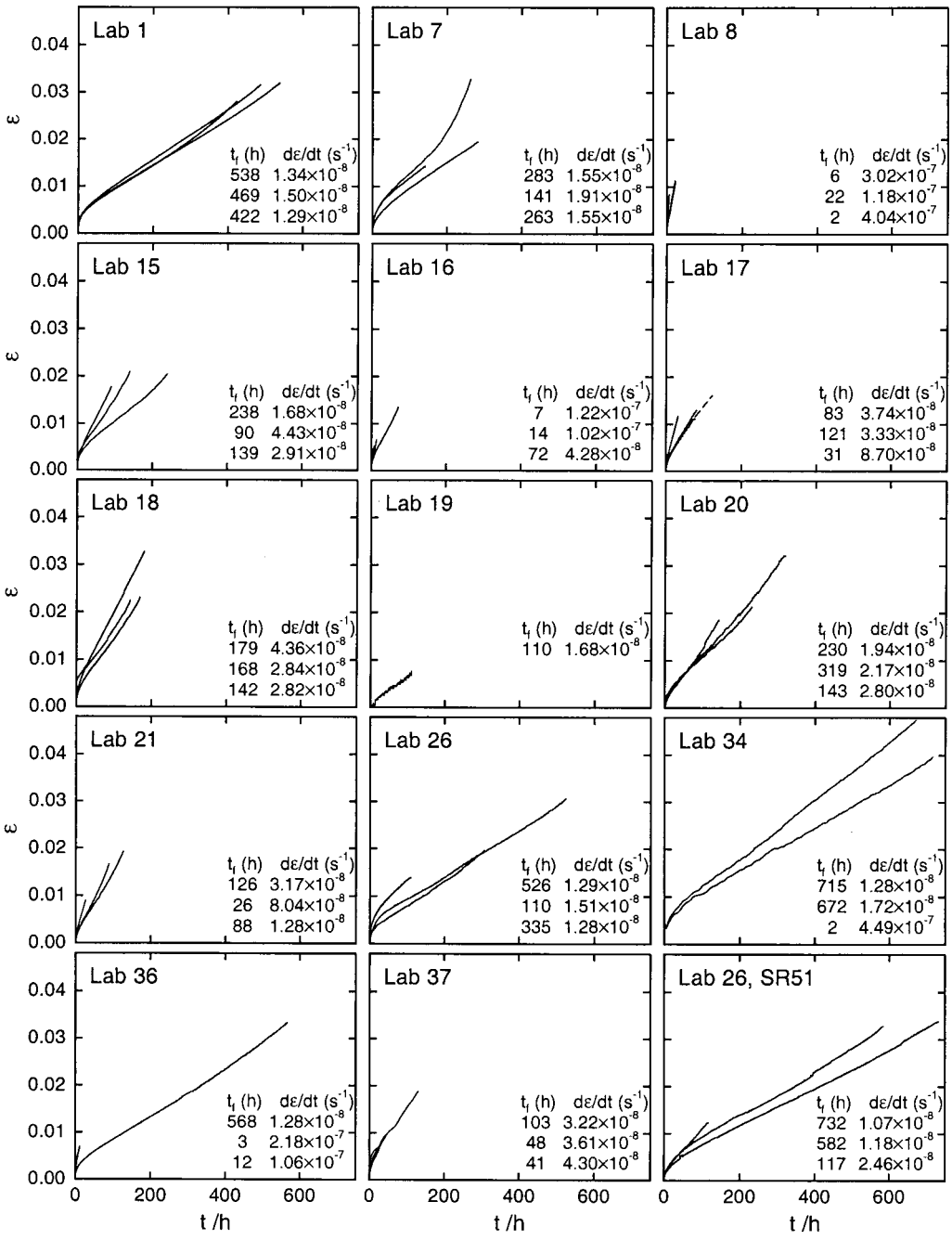

Fig. 2. Fifteen sets of creep curves for this study. Each subfigure lists the times to failure and minimum creep rates for the specimens.

strains to failure, not tabulated on the graphs, vary by a factor of 4. None of these parameters correlated with billet of origin. Because time to failure and minimum creep rate of silicon nitride depend exponentially and by power law on temperature and stress, respectively, Fig. 3 uses logarithmic axes for these plots. Plotting this way allows direct comparison of the relative variability within a given laboratory. Note that, while some laboratories had very repeatable results, most notably laboratories 1 and 18, the scatter for some other laboratories was quite large.

The fracture surfaces of all the specimens, whether large or small diameter, or large strain to failure or small, showed the typical rough surface normally associated with final failure by crack growth under creep conditions. (See, for example, micrographs of other silicon nitrides in Refs. 11 and 17–21, and especially the schematic diagram in Ref. 19.) In all but three specimens, the rough region associated with the crack growth intersected the surface. The failure times, strains, and creep rates of the three specimens failing from internal creep crack growth were not uncharacteristic of the full group, however.

IV. Discussion

The most important result of this study is the observation that, even for a well-behaved material, the creep parameters measured in competent laboratories can differ substantially. Taken individually and evaluated in terms of repeatability, no one laboratory stands out from any other. The laboratory results would have been unconditionally and correctly accepted as representative by any reasonable consumer of tensile creep data. Certainly laboratories 34 and 36 would have discounted their obviously premature failures in any formal report. Excluding these, the mean times to failure range from 10 h (laboratory 16) to 476.4 h (laboratory 1): a factor of almost 50. The minimum creep rates vary by almost 20

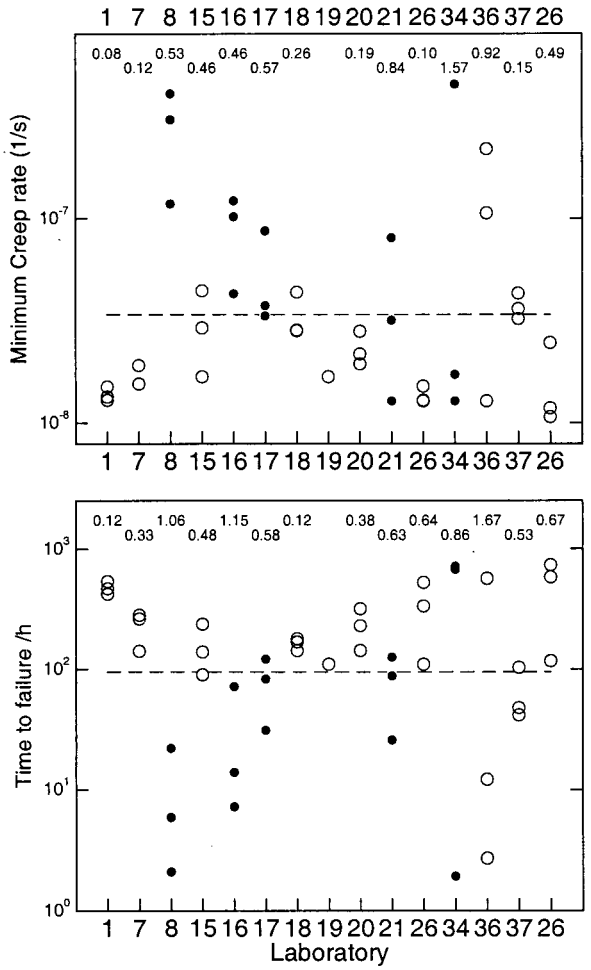

Fig. 3. Time to failure and minimum creep rate for the 14 laboratories. Dashed lines represent the mean values of the natural logarithms. Numbers above the data points are the relative standard deviations, that is, the sample standard deviation divided by the mean value. Right-most entry for Laboratory 26 corresponds to the tests on the SR51 specimens. Solid symbols represent tests on the large-diameter, buttonhead specimens and open symbols represent tests on small cross-section specimens.

times. Of course, the very small strains to failure for laboratory 8 may represent only primary creep. In hindsight, one could argue that the measured creep rate might have decreased further given sufficient time, but nothing about the creep curves of laboratory 8 indicate that simply choosing the minimum creep rate, which is a common practice in tensile creep studies of silicon nitride, would not be valid.

If we eliminate the extreme short-time failures in laboratories 34 and 36, the within-laboratory variability in time to failure and minimum strain rate is fairly constant across the laboratories: Table II summarizes the individual values from Fig. 3, as well as those from other studies of tensile creep of silicon nitride. The predecessor to this study,[5] in which five laboratories tested identical specimens of an earlier vintage of the material of this study, as well as a parallel study by French and Wiederhorn[14] demonstrated that very high reproducibility and repeatability are achievable. In the five-laboratory study, no laboratory showed more than 20% relative standard deviation on any measurement. However, the variability in creep parameters of two other commercial silicon nitrides[18,22] was similar to that encountered in this study (see Table II).

(1) Sources of Variability

Variability in the measured creep parameters, both within- and between-laboratory, can result from exogenous and endogenous

Table II. Repeatability of Tensile Creep of Silicon Nitride

Specimen	Relative standard deviation		
	ε_f	t_f	$d\varepsilon/dt]_{min}$
This study			
All laboratories	0.07–1.09	0.12–1.67	0.08–1.57
Obvious premature failures removed[†]	0.07–0.53	0.12–1.14	0.08–0.84
Menon *et al.*[18] (buttonhead)			
1371°C, 145 MPa (17 tests)		0.58	0.42
1371°C, 180 MPa (6 tests)		0.62	0.37
Wereszczak and Luecke, reported in Ref. 22, 1275°C, 137.5 MPa			
SR76 (7 tests)	0.14	0.24	0.22
DR51 (9 tests)[‡]	0.16	0.42	0.41
Buttonhead (4 tests)	0.08	0.45	0.67
French and Wiederhorn,[14] 1300°C, 250 MPa, 5 tests on each specimen			
SR30[§]	0.14	0.07	0.06
SR51	0.10	0.12	0.07
SR76	0.11	0.07	0.04
French and Wiederhorn,[14] 1400°C, 150 MPa, 5 tests on each specimen			
SR30	0.08	0.12	0.09
SR51	0.06	0.13	0.10
SR76	0.15	0.10	0.14
Initial round-robin, SR76,[5] 1400°C, 150 MPa			
Laboratory 1 (4 tests)	0.14	0.14	0.05
Laboratory 3 (4 tests)	0.07	0.07	0.19
Laboratory 4 (4 tests)	0.10	0.10	0.10
Laboratory 6 (5 tests)	0.12	0.12	0.15
Laboratory 7 (4 tests)	0.11	0.02	0.09

[†]Laboratories 34 and 36. [‡]DR51 specimen is 51 mm long and similar to the specimen of Ref. 7, but with a second reduction in gauge width. [§]SR30 specimen is 30 mm long with 1.5 mm × 2.0 mm cross section.

sources. Deviations from the desired temperature and stress (i.e., exogenous contributions to variability) can produce quite measurable changes in creep rate and failure time. From the expressions for creep rate and time to failure developed for an earlier vintage of the material of this study tested at NIST,[15] the creep rate increases 2.6%/MPa and 3.3%/K, while the failure time decreases 3%/MPa and 3%/K in the neighborhood of the round-robin test conditions. To explain entire between-laboratory variability of mean failure times would require that the conditions in the extreme laboratories differed by either 127°C or 130 MPa. For the minimum creep rates, the deviations are similarly outrageous: 47°C and 58 MPa. The typical within-laboratory variability in time to failure or creep rate would require a temperature range of almost 20°C between tests, which is an unreasonable expectation. Clearly, temperature and stress deviations cannot be the sole source of either the between- or within-laboratory variability. Although the creep community (following the lead of the metals community[23]) very reasonably believes that poor alignment can reduce time and strain to failure,[24] there have been no specific studies correlating the degree of misalignment with creep rupture lifetime in structural ceramics. Because the instructions for the round-robin did not specify that the laboratory quantify the degree of bending in the specimen, it is not possible to assess the effect of bending on creep rate or lifetime. However, several of the laboratories that reported rather sophisticated alignment procedures also reported more than average variability in creep parameters. In general the degree of care in alignment reported by the laboratories did not correlate with the variability of the rupture lifetime. Absolute strain measurement errors probably do not contribute significantly to the observed variability in creep rates. Contact extensometry should provide quite high accuracy and precision,[25] and flag-based extensometry typically produces strains that are accurate to 10%.[8]

In addition to exogenous sources of variability, over which the testing laboratory has nominal control, there are several potential endogenous (that is, resulting from the material itself) sources. Specimens taken from different billets, or even different locations in the same billet, may have subtle chemical and phase differences

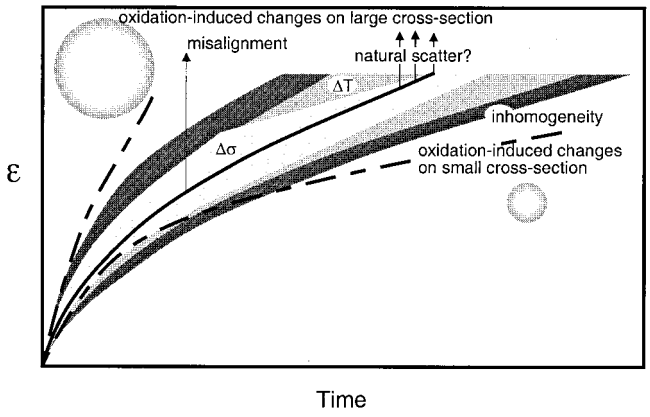

Fig. 4. Ways in which deviations from ideal experimental conditions may affect creep of Si_3N_4.

that can alter their creep behavior. Although the creep community has invoked the so-called vintage effect to explain differences between laboratories or billets for decades,[26,27] there are no published comparisons quantifying the sources or potential magnitude of the variability beyond the assertion that they exist. Finally, despite several theoretical[28,29] and experimental studies[11,30] of creep rupture of structural ceramics, it remains unclear if time and strain to failure are stochastic or deterministic quantities for silicon nitride, or even if failure originates from damage accumulation or from preexisting flaws.

All these overlapping sources of variability, illustrated schematically in Fig. 4, can affect an individual laboratory, and make a more substantial contribution to the differences between laboratories. The mixing of these effects complicates analysis of the causes of the within- and between-laboratory variability and restricts discussion to only very coarse trends.

(2) Specimen Size Effects

The wide variety of specimens, gripping schemes, extensometry techniques, and temperature measurement strategies makes definitive assignment of the root causes of between-laboratory variability perilous and complicated. Of the many possible comparisons, however, one stands out as particularly significant and compelling. As a group, the small cross-section specimens lasted about 5 times longer and crept about 3 times slower than the large cross-section buttonhead specimens (see Table III).

There are two possible sources for such behavior. One major difference between the pin-loaded specimens and the buttonhead specimens is that the latter generally have a small, but measurable, temperature gradient in the gauge length, caused by heat conduction out the ends of the specimen into the water-cooled grips. Most of the laboratories testing buttonhead specimens have attempted to ameliorate this problem by considering the average temperature on the gauge length, typically obtained by cementing thermocouples onto, or sometimes into,[22] dummy specimens. All reported <12°C difference between the hottest and coldest regions of the gauge length, centered on the desired test temperature. Using the expression for minimum creep rate of the earlier vintage of the material of this study developed in Ref. 15, that deviation, applied to the entire gauge length, an extreme case, would only increase the creep rate about 1.5 times, rather than the factor of 3 observed.

Table III. Comparison of the Creep–Rupture Parameters of Small- and Large-Diameter Specimens

Parameter	Large specimens†	Small specimens†
Number of specimens‡	14	25
Mean $\log_e t_f$ (h)	3.77	5.36
Mean t_f (h)§	43	213
Variance of $\log_e t_f$	2.86	0.61
Mean $\log_e d\varepsilon/dt]_{min}$ (1/s)	−16.65	−17.71
Mean $d\varepsilon/dt]_{min}$	5.88×10^{-8}	2.04×10^{-8}
Variance of $\log_e d\varepsilon/dt]_{min}$	1.16	0.20

†All small-diameter specimens have cross-sectional area $<1.2 \times 10^{-5}$ m^2; most are $<6.5 \times 10^{-5}$ m^2. All large-diameter specimens are buttonheads, with cross-sectional areas $>2.8 \times 10^{-5}$ m^2. ‡Obvious premature failures from Laboratories 34 and 36 removed. §Mean values are calculated on the natural logarithms of data and then exponentiated, to allow pooling of variances.

A more plausible source for the difference between large and small specimens lies in their response to the oxidizing conditions during the test. It is a nearly universal phenomenon that annealing silicon nitride in air improves its creep resistance.[31–40] Only rarely does it degrade creep resistance.[38,41] The surface oxide layer that forms during oxidation is only one of the many responses of the material. The chemistry and types of the minor phases, as well as the chemistry of the amorphous, siliceous layer on two-grain boundaries,[42] can change up to several millimeters below the surface.[43–49] Frequently these changes manifest themselves as rings of different-colored material[48,50–54] that are absent for specimens tested in argon,[55] changes in hardness,[48,51] changes in the distribution of second-phase cations,[43–47,53,56,57] changes in the thickness of the amorphous, siliceous film on two-grain boundaries,[42] as well as changes in the secondary[22,58] and silicon nitride phases.[11] Although the exact mechanisms of deformation are not resolved, researchers generally agree that the nature of the second phase controls the tensile creep rate in silicon nitride.[59–62] Time-dependent changes to the second phase may alter the creep response dramatically and may cause part of the primary–secondary–tertiary creep response that silicon nitride so often exhibits.

If these changes are limited by diffusion of oxygen through the oxide scale and down the silicon nitride triple junction network into the bulk of the specimen[11,50–53] or by diffusion of second-phase cations out of the specimen, as most of the oxidation literature suggests,[43,44,56,63–66] then the oxidizing atmosphere affects the bulk of the small-diameter specimens more rapidly. Figure 4 attempts to illustrate how equally sized reacted zones affect the small specimen much more strongly. The buttonhead specimen is primarily unaffected, while the small-diameter specimen is mostly transformed. If the reacted zone is more creep resistant than the unreacted material, a gradient in stress results across the diameter, and the predominantly unaffected buttonhead specimen creeps faster than the smaller specimens.

The research community generally agrees that out-diffusion of sintering aids, in response to the activity gradient caused by the pure silica formed, controls oxidation of silicon nitride.[43,44,56,66] However, demonstrations of the formation of second-phase concentration gradients during oxidation generally have used conditions much more severe than those of typical creep experiments; therefore, this mechanism may not be relevant to the changes in creep response during oxidation. For example, oxidation of a commercial, magnesia hot-pressed silicon nitride for 8 h at 1400°C produced a 300 μm deep zone depleted in magnesium.[43] In contrast, the most severe published tensile creep test on this material was at 1260°C, under a 103.3 MPa load, which resulted in failure in <10 h.[67] Similarly, oxidation of a ytrria hot-pressed silicon nitride for 16 h at 1495°C produced a zone depleted in yttria only 15 μm deep. In creep rupture, a similar vintage of this material failed after only 1 h at 100 MPa at 1300°C.[68] There are very few demonstrations of significant cation-depleted zones in crept specimens. Chartier and Besson[53] found a surface zone depleted in cations whose size correlated with the macroscopically visible colored rind extending in from the specimen surface. Besson *et al.*[69] found a 400 μm deep zone depleted in yttrium in a Y_2O_3–Al_2O_3–Si_3N_4 crept for 50 h at 1350°C. Lofaj,[57] using microfocus X-ray tomography, found a zone depleted in ytterbium ~600 μm deep in a Yb_2O_3–Al_2O_3–Si_3N_4 crept for 263 h at 1400°C. That specimen had a 10 h primary creep regime over which the creep rate decayed to ~5% of its initial value.

Diffusion of oxygen into the silicon nitride via the second-phase triple-junction network may alter the nature of the second phases, and with it the creep response. There has been little published confirmation of this mechanism, although Wereszczak *et al.*[11] did investigate the effect of the oxidizing atmosphere on the failure of

silicon nitride. Wereszczak *et al.*[22] also recently found that special buttonhead specimens of an earlier vintage of the material of this study with diameters 2 mm × 4 mm crept about one-half as fast as those of the normal 6.35 mm diameter and that the slightly decreased average specimen temperature could not account for the decreased creep rate. During exposure, Yb_2SiO_5 replaced $Yb_2Si_2O_7$ and $Yb_2Si_2N_2O_7$ as the second phase. Similar creep behavior occurred in unpublished comparison of another commercial, hot isostatically pressed silicon nitride:[22] large-diameter buttonhead specimens tested at the Oak Ridge National Laboratory High Temperature Materials Laboratory (ORNL–HTML) crept much faster and failed sooner than smaller-cross-section pin-loaded specimens from the same billets tested at NIST. Static annealing at the test temperature for 196 h before testing extended the failure time of the buttonhead specimens to approximately that of the pin-loaded specimens. All the specimens had obvious millimeter-thick rinds of much darker color at the specimen surface.

(3) Implications for Tensile Creep Rupture Testing of Si_3N_4

Unfortunately, this interlaboratory study did not unequivocally implicate one single cause of either the within- or the between-laboratory variability. However, we can combine the experiences from this study with several other informal interlaboratory comparisons[5] to make some recommendations for tensile creep rupture testing of silicon nitride. The most important recommendation is that casual temperature and stress measurements are unacceptable. Because the creep response of silicon nitride is so sensitive to temperature and stress, typically 3%/K, researchers must have great confidence that the temperature that their thermocouples indicate is truly the temperature that their specimens experience. This requirement means that the thermocouple must return repeatedly to the same position in the furnace from test to test, perhaps to within millimeters. The thermocouples must not drift over time because of contamination or diffusion: regular calibration is essential. Finally, formal reports must clearly communicate the means by which quality of the stress and temperature measurements were assured. If it is suspected that oxidation-induced changes alter the creep response, then preannealing the specimen at the highest test temperature for longer than the longest expected test may stabilize the microstructure and minimize the effect. This approach should be used with care, because the creep properties of the resulting material may not resemble those of the starting material. More interlaboratory testing is probably not warranted until the sources of variability are more clearly identified and strategies to eliminate them are developed. Before initiating any new interlaboratory study, it will be essential to verify that the candidate material has highly repeatable creep-rupture properties.

V. Summary

(1) Consumers of silicon nitride tensile creep data must accept the fact that data from competent laboratories may differ by an order of magnitude or more from equally acceptable data produced in other competent laboratories.

(2) This study revealed a specimen size effect on time to failure and minimum creep rate. The large-diameter, buttonhead specimens failed in about one-fifth the time, and had creep rates about 3 times larger than smaller-diameter pin-loaded specimens.

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